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Characterization of composite hydrocolloid film based on sodium cellulose sulfate and cassava starch

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ABSTRACT

The objective of this study was to investigate the effect of the starch incorporation and homogenization temperature on the properties of cellulose sulfate (NaCS)-based films plasticized with glycerol. The film-forming solution, physicochemical microscopic and mechanical properties of films were studied. The SEM of the composite film was more rough than NaCS films, but no signs of phase separation between components were observed. As the starch proportion increased, composite films became more fragile to break, whereas a positive effect on the elasticity of films was realized by a considerable increase in *E*% values. The composite films presented lower water vapor permeability than films without starch. The temperature for preparation of film-forming solution also had adverse effect on film properties. NaCS film incorporated with 25% starch showed greatly improved resistance to water, though 5% decreasing in TS and small change in other properties compared with NaCS film.

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1. Introduction

Environmental concerns enhance and stimulate the use of renewable resources as edible film for packaging to maintain or even improve life quality of foods (Garcia et al., 2004). Usually, edible films that can help to maintain moisture and improve shelf life of coated foods are made of proteins or polysaccharides (Vu et al., 2011). Specifically, several polysaccharides, such as cellulose derivatives, chitosan, starch, alginate, carrageenan and pectin, are important candidates for their good film forming capacities.

Cellulose derivative-based films have the obvious superiority in raw material resource and mechanical property. Commonly cellulose derivatives used for preparation of edible films include carboxymethyl cellulose (CMC), methyl cellulose (MC), hydroxypropyl cellulose (HPC) and hydroxypropyl methyl cellulose (HPMC), which have the advantages of suitable strength, good flexibility and transparency, resistance to oil, as well as water solubility, colorless and tasteless properties, etc. (Bourlieu et al., 2009; Debeaufort et al., 1998; Johansson et al., 2012; Schmitt et al., 1998). Sodium cellulose sulfate (NaCS) is a novel cellulose derivative containing the active group of $-SO_3^-$, which has some ideal properties such as biocompatibility, biodegradation, water-soluble, and good film forming behaviors (Chen et al., 2013; Mei and Yao, 2002). At the same time, various preclinical studies show that NaCS possesses significant antivirus activity especially for X4 tropic and R5 tropic HIV (Neurath et al., 2002; Scordi-Bello et al., 2005). Therefore, NaCS becomes a new candidate for preparation of edible film because of simple preparation process, low cost and being suitable for large-scale production.

To improve or regulate the properties of edible film, the polysaccharide, protein or lipid are generally mixed to fabricate composite edible films. Starch was often incorporated to edible films prepared from different materials due to its abundance in nature and cheap price. Some edible films based on mixtures of starch and other materials such as starch-methylcellulose, pullulanstarch, chitosan-starch and CMC-rice starch have been investigated. The effects of starch addition on kefiran/corn starch composite films' physical, mechanical and water-vapor permeability (WVP) properties were investigated by Motedayen et al. (2013). Corn starch-carboxymethylcellulose/methylcellulose biodegradable films were prepared by Kibar and Us (2013). The effect of gelatin concentrations on the mechanical properties and solubility of cassava starch-based films containing glycerol was studied by Tongdeesoontorn et al. (2012). Some work aimed to study the effect of starch and maltodextrin on the properties of soy protein isolate (SPI) films (Galus et al., 2012).

However, there is negligible data available about the physicochemical properties of NaCS-starch composite films. The present study was aimed at investigating the effect of cassava starch content on properties of NaCS-based edible films and the potential usage of NaCS-starch films as packaging material. The homogenization temperature of film-forming solution was also studied to assess its influence on mechanical properties and water vapor permeability.





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2. Materials and methods

2.1. Materials

Sodium cellulose sulfate (NaCS) with dynamic viscosity ($\eta_{2\%}$, 2 wt% solution) of 535 mPa s⁻¹ was prepared by heterogeneous reaction in our lab (Chen et al., 2013). Food grade cassava starch (ST) was obtained from JUXIANG Starch Co., Ltd (Xiamen, China). Analytical grade glycerol as plasticizer was purchased from Sinopharm Chemical Reagent Co., Ltd. (China). Distilled water was used for all sample preparations.

2.2. Preparation of edible films

Films were prepared according to the so-called casting technique. An aqueous solution of NaCS (2%, w/w) was prepared with 2 g NaCS dissolved in 98 g distilled water with continuous stirring at 200 rpm for 30 min at room temperature, followed by degasification. ST solution (2%, w/w) was prepared with 2 g starch dissolved in 98 g distilled water with continuous stirring at 200 rpm for 60 min at 90 °C followed by cooling until room temperature, and then degasification. The NaCS-ST composite films forming solution was obtained by blending NaCS solution, ST solution and glycerol according to Table 1. The effects of ST content (0%, 15%, 25%, 35%, 50%, 75%, 100% on the total solid mass) and homogeneous temperature (30 °C, 40 °C, 50 °C, 60 °C, 70 °C) as shown in Table 1 on film forming were investigated. After cooling and degasification of film-forming mixture, each of the samples (26 ml) was poured into an even surface Teflon mold (diameter = 12 cm), dried at 60 °C for 4 h, and then carefully peeled off from the casting surface and conditioned for 24 h at 25 °C and 75% RH in desiccators with a saturated NaCl solution prior to further analysis. All the design points had three replications.

2.3. Viscosity measurements of film-forming solution

Flow curves of film-forming solutions were recorded at 30 ± 0.5 °C with a NDJ-5S rheometer (model NDJ-5S, Shanghai Fangrui Instrument Co. Ltd., China), using a coaxial cylinder device. The range of shear rate, $100-300 \text{ s}^{-1}$, was used because it covered all the concentrations using the same cone plate geometry. The rheometer was connected to a thermostatted bath. Viscosity was calculated by dividing shear stress by shear rate. Each solutions were analyzed in triplicate.

2.4. Thickness measurements of film

The samples were conditioned at 25 °C and 75% RH (a saturated NaCl solution) for 24 h to determine the thickness, and the thick-

Table 1	
Experiment	design

Sample	Starch content in total solid mass" (%)	Film-forming Solution Composition			
		2% (w/w) NaCS Solution (g)	2% (w/w) Starch Solution (g)	Glycerol (g)	Temperature (°C)
S1	0	100	-	0.3	30
S2	15	85	15	0.3	30
S3	25	75	25	0.3	30
S4	35	65	35	0.3	30
S5	50	50	50	0.3	30
S6	75	25	75	0.3	30
S7	100	-	100	0.3	30
S8	25	75	25	0.3	40
S9	25	75	25	0.3	50
S10	25	75	25	0.3	60
S11	25	75	25	0.3	70

* The total solid mass of film is the sum of dry NaCS weight and dry starch weight.

ness of the samples was measured (exactness of ±0.001 mm) using a digital external micrometer (Mitutoyo Co., Japan) at ten different points of the film. The mean values were used in the calculation of mechanical properties and WVP.

2.5. Mechanical tests

Tensile strength (TS) and elongation at break (*E*%) of the films were measured according to standard method (ASTM, 2001) using a Instron Universal Testing Machine (Instron Corp., model 5569, U.S.A.). Test samples, 25×100 mm, were cut from each film and fixed on the grips of the device with a gap of 30 mm. They were then pulled apart at crosshead speed of 20 mm/s and preload of 2 N. The average thickness of films was $25 \pm 2 \mu$ m. Three replicates of each film were tested. TS(MPa) was calculated by the following Eq. (1):

$$TS = F_{max}/S \tag{1}$$

where F_{mas} is the maximum force (N) needed to pull the sample apart; A is the cross sectional area (m²) of films. *E*% is calculated based on the following Eq. (2):

$$E\% = \Delta L/L_0 \times 100\% \tag{2}$$

where ΔL is the film elongation at the moment of rupture (mm) and L_0 is the initial length of samples.

2.6. Water vapor permeability tests (WVP)

Water vapor permeability of the films was measured using the standard ASTM method (1995) (ASTM, 1995). The films were cut into 90 × 90 mm pieces and each piece was put onto a permeability cup. The cup was previously filled with fused anhydrous CaCl₂ (RH = 0%). The cup was then sealed with a cover and put into a humidity chamber at 25 °C and 75% RH for 3 days. The sealed cup was weighed periodically (±0.0001 g). Water vapor transport into the cup was determined by the weight gain of the cup. Linear regression was used to fit the data, weight vs. time, and to calculate the slope of the plot. The water vapor transmission rate (WVTR) is calculated from the slope $\Delta w / \Delta t$) of the straight line divided by the test area (A) as Eq. (3), (g s⁻¹ m⁻²).

$$WVTR = \Delta w / (\Delta t \cdot A) \tag{3}$$

where Δw is the weight of water absorbed in the cup (g), Δt is the time for weight change (s), *A* is the area exposed to moisture transfer (m²). The WVP (gm⁻¹ s⁻¹ Pa⁻¹) is calculated as Eq. (4).

$$WVP = (WVTR \times L)/\Delta p \tag{4}$$

where *L* is the thickness of the film (m) and $\triangle p$ is the difference of partial water vapor pressure across the film ($\Delta p = p(RH_2 - RH_1) =$

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